

## **Scintag XRS2000 Powder Diffractometer Operation Summary**

*02/14/2012 A.E.*

**Location:** 1709 Gilman Hall

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### **Safety**

All users of Scintag XDS2000 must complete the EH&S “X-ray Safety Fundamentals” course available on-line at [www.ehs.iastate.edu](http://www.ehs.iastate.edu). The valid “X-ray Safety Fundamentals” Certificate is mandatory to enter 1709 Gilman Hall X-ray lab.

This class demonstrates the correct procedures for the authorization and use of X-ray producing devices. Topics include X-ray production and hazards, hazard control measures, and regulatory requirements. Certification is awarded upon successful completion of the quiz at the end of the course. You must recertify every year.

All diffractometer enclosures in CIF X-Ray Laboratory are checked by EH&S annually and certified for data collection operations. All systems are also equipped with multiple interlock switches blocking the X-ray shutter to avoid any possible operator exposure.

### **Training Requirements**

Before operating diffractometer, you **MUST** be properly trained and authorized by CIF Supervisor of the X-ray lab!

This training manual includes information about the diffractometer and step-by-step instructions on sample preparation and data collection.

All referenced documents, including the detailed “DSMT Software Manual” and the “User’s Manual” are available in 1709 Gilman Hall.

## 1. Sample Preparation:

1.1. Place significant amount of a sample into the mortar and grind with pestle by strong circular motion for producing small particles. The powder should consist from more or less even particles.

1.2. Use a spatula to move powder into sample holder. Take a holder which fits to amount of sample you have. The larger area/amount of powder you have – the better intensity you can observe.

1.3. There is a small sample holder (111- oriented single crystalline plate of quartz) in a designated drawer. This is the best holder for small amounts of substance. Moreover, this holder is the best possible solution for precise measurements as it has ZERO intensity at all diffraction angles!!! **Such a holder costs \$1200 dollars and there is only one available, please, be very careful when using it. Clean it after data collection and put back to the drawer. It is strictly prohibited to take this slide out of 1709 Gilman!!!** If you need to prepare sample in your lab you MUST request the reservation of this slide and an instrument in advance. Please, e-mail requests at [ellern@iastate.edu](mailto:ellern@iastate.edu)

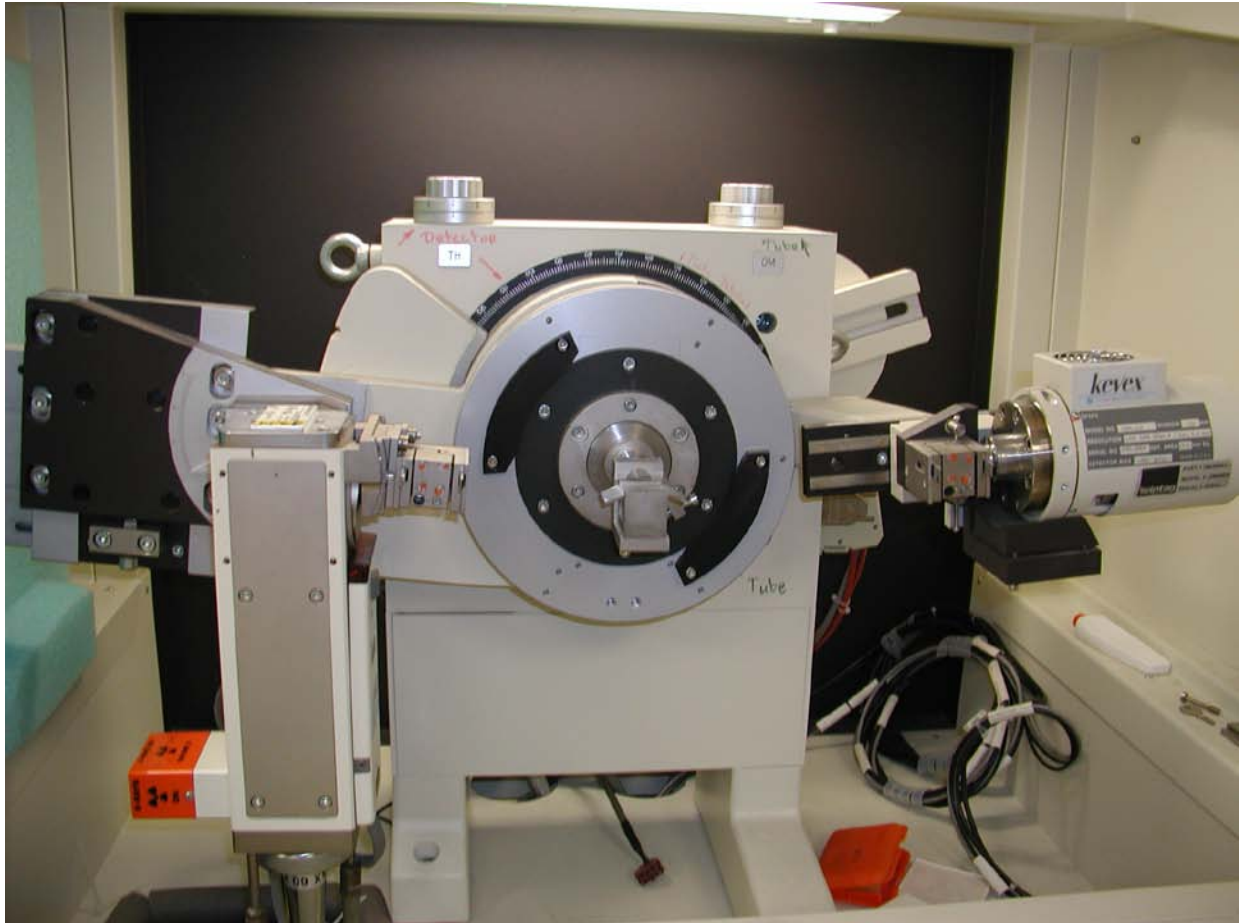
1.4 Take a clean microscope glass slide and press the pellet flat. You can use some circular movements with small pressure to flatten the surface. Sometimes the dry powder sticks to the glass because of static electricity. The standard procedure for such samples is to mix some mineral oil with the powder. Do not forget, that all Mineral Oils/Vaseline have broad peaks at some angles and you should correct the final powder pattern.

1.5. Please, clean the microscope glass with ethanol and tissue and put it to the special container for the used glasses. Those glasses are not disposable and they are expensive.

1.6. Please, clean the desk after your work. The safety goggles, respirators, latex and plastic gloves can be found on a lab desk or on a shelf above the desk. Please, use all

necessary precautions when you are working with your samples. If you need some other supplies for your work, please, e-mail at [ellern@iastate.edu](mailto:ellern@iastate.edu)

## 2. Startup Procedure:



**Fig. 1. SCINTAG XDS2000 Goniometer. The tube housing with conventional Cu-radiation source is on the left side of and the KEVEX detector -on the right side of goniometer.**

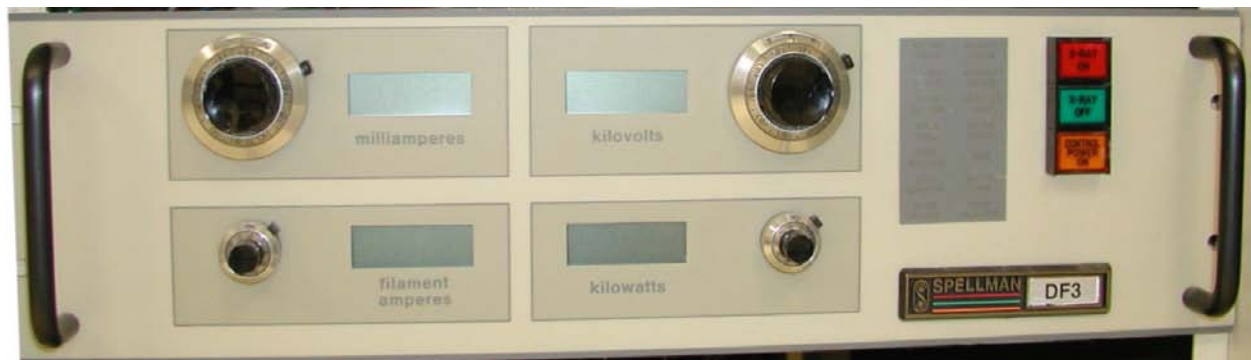
2.1. Begin by checking settings of angles of X-ray tube and detector. Both Detector (Theta) and Tube (Omega) angles have to be preset to 5.0 Degrees – that means 5 degrees on the integer scales and 0 on the Vernier scales. **If the values are different from 5.0, please, e-mail at [ellern@iastate.edu](mailto:ellern@iastate.edu)**



**Fig. 2. Water and interlock switches.**

2.2. Open sample chamber and insert sample. Close chamber and press **Interlock Reset** button to turn off light (Fig. 2). **This button MUST be dark during all further operations!!!!**

2.3. Turn red **Water** switch **ON** – Light should now be lit (Fig. 2)



**Fig. 3. Spellman X-ray Generator**

2.4. Press orange switch **Control Power On** – Light should now be lit during all further operations (Fig. 3).

2.5 Press red switch **X-ray On** – Light should now be lit for data collection (Fig.3).

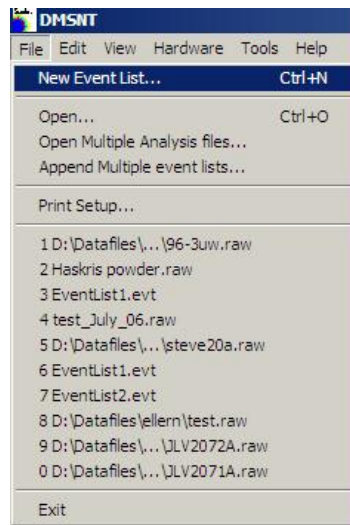
2.6. Wait for generator to reach default values of -44 kV / 28 mA.

### 3. Data Collection. Operating Procedure:

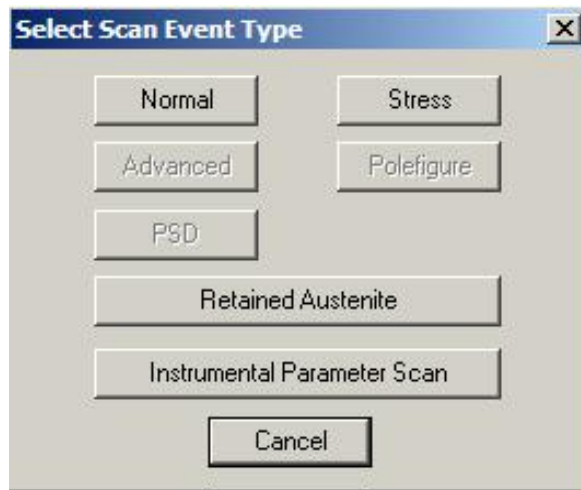
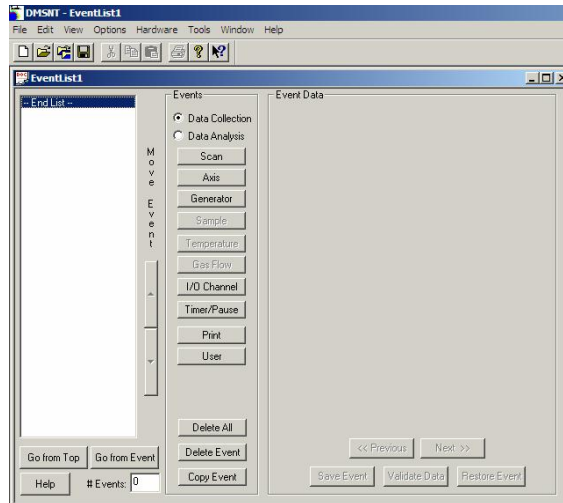


3.1 Log onto computer and DMSNT program

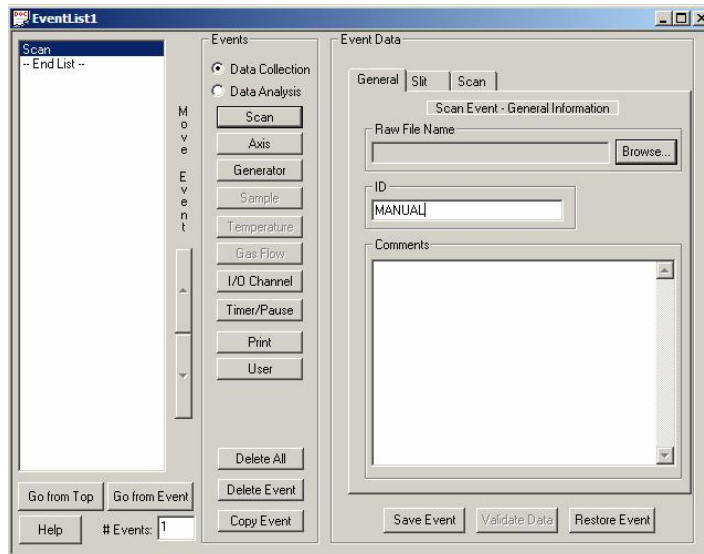
3.2. File → New Event List



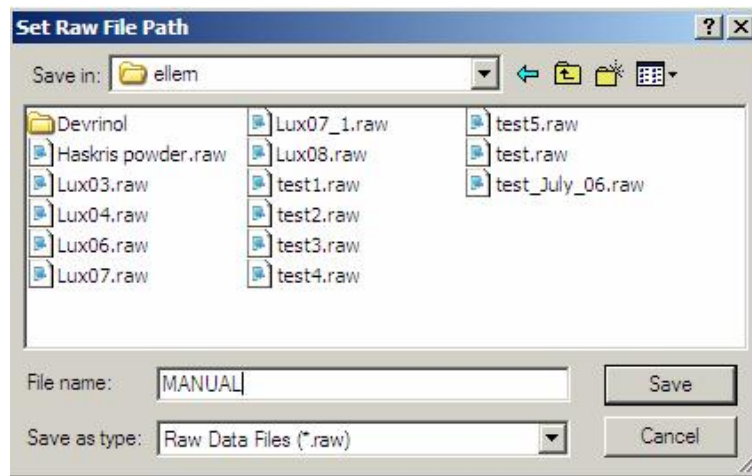
3.3. Scan → Normal



### 3.4 Create a 'Sample ID'



3.5. **Browse** → and go to your directory on E:/Datafiles/YourLOGINNAME and create a name for output file

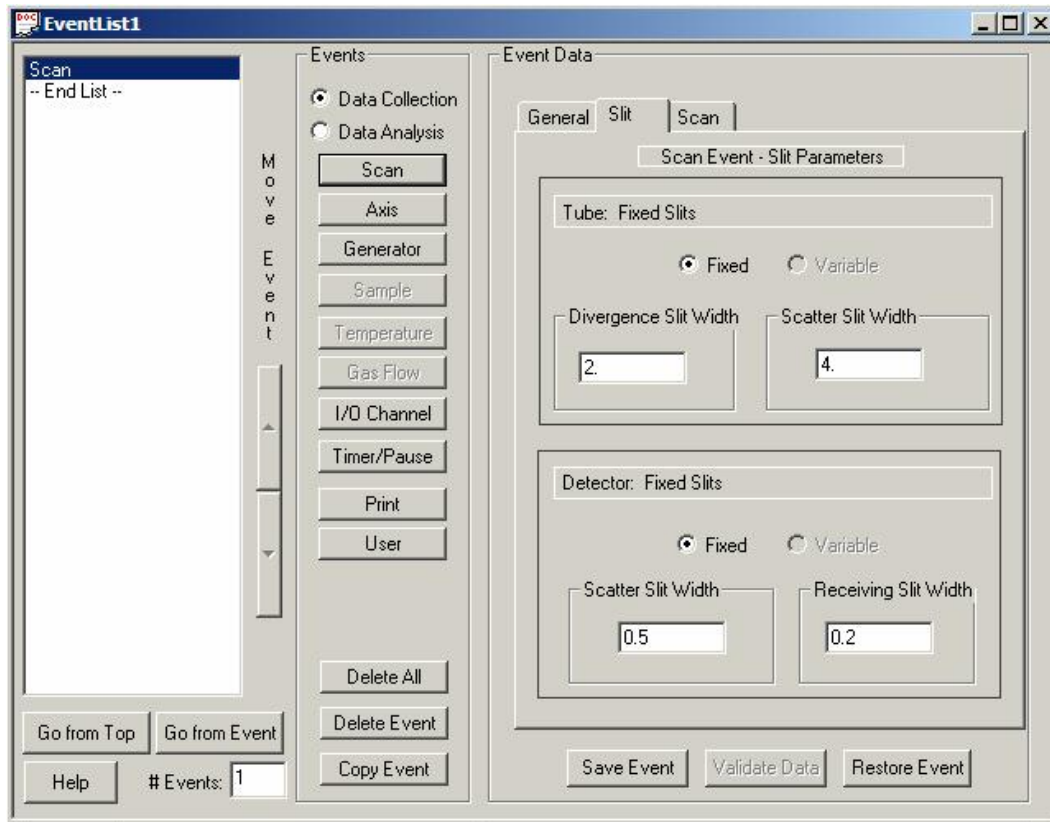


3.6. Do to the **Slit** Tab and check the slit values. The default values are **2** and **4** mm on tube housing and **0.5** and **0.2** mm on a detector (Fig. 4). If you need to change slits for your measurements, do not forget to change the values accordingly. You have to place default slits back when you will be done with your measurements.

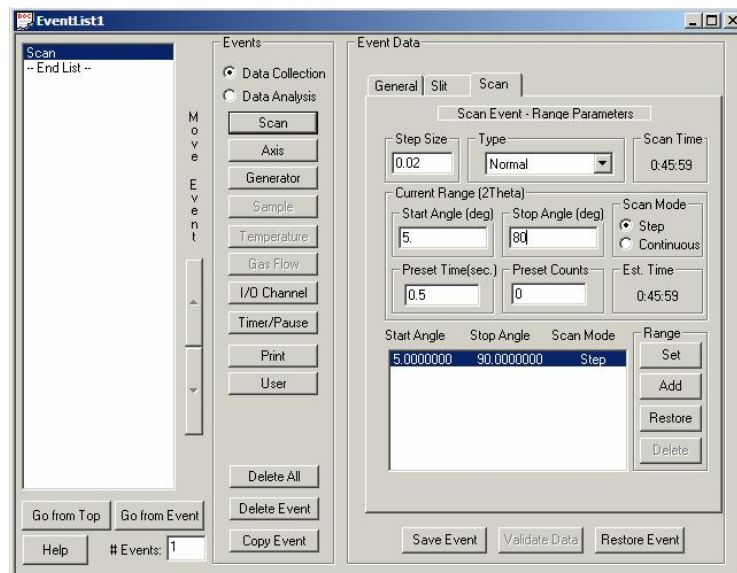


**Fig. 4. The slits positions on a X-ray housing and detector.**





### 3.7. Go to Scan Tab



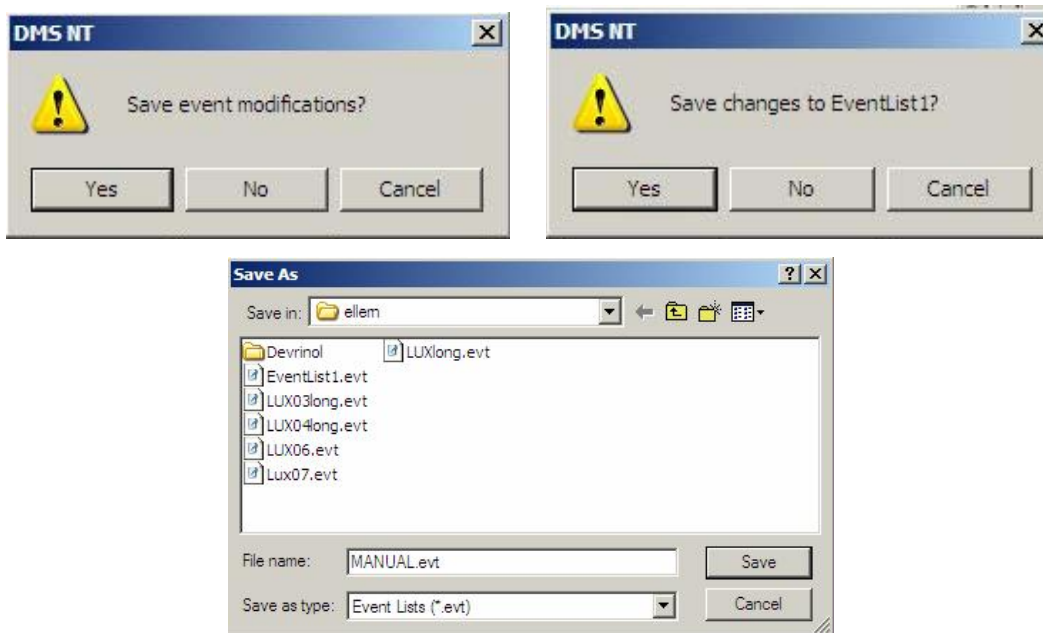
Set 'Step Size' ( default) 0.02 degrees

Define **Start Angle** and **Stop Angle** limits (**1° is a minimum!!!**)

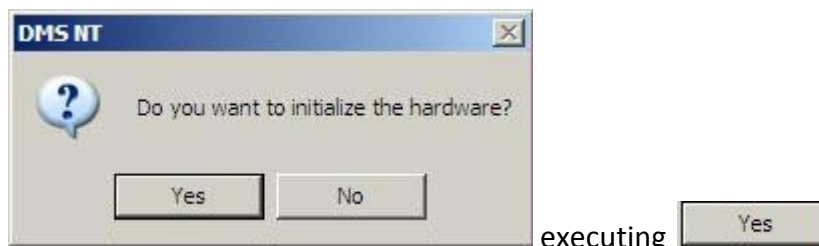
You have to use the **Set** button to save all changes

3.8. Execute **Go from Top** button to begin data collection.

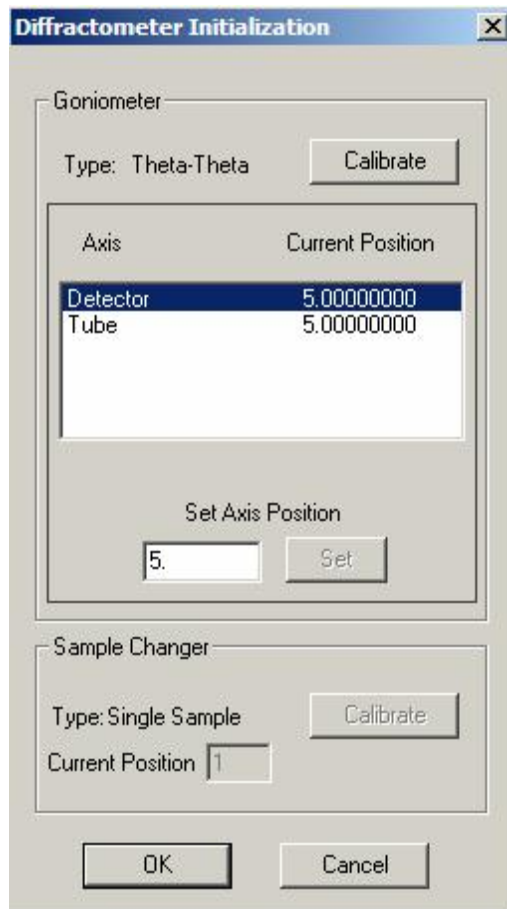
Follow on screen prompts to save files with the data collection settings.



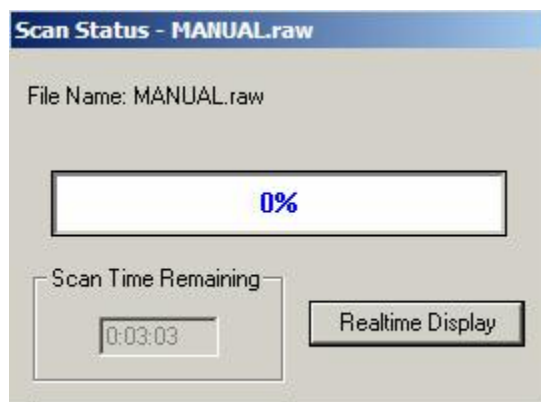
3.9. Initialize Hardware



Now the computer connects to the goniometer controller and you should see the “**Diffractionmeter Initialization**” screen and the values for both Detector and Tube **MUST BE 5.0 degrees**. . If the values are different from 5.0, please, e-mail at [ellern@iastate.edu](mailto:ellern@iastate.edu)

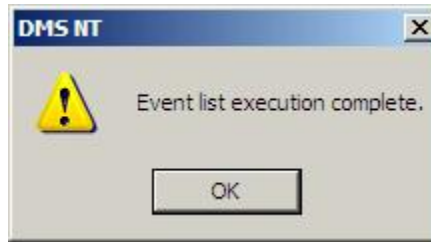


3.10. Push  to begin data collection.



If you want to monitor data collection, use  button.

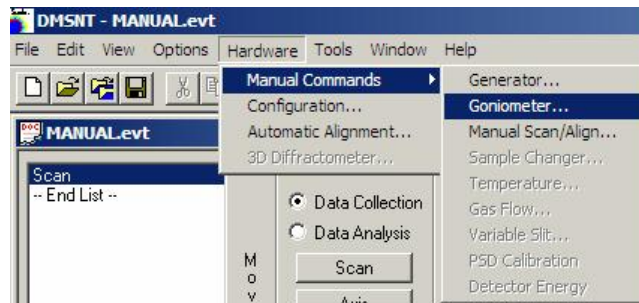
3.11. After data collection is complete you will see



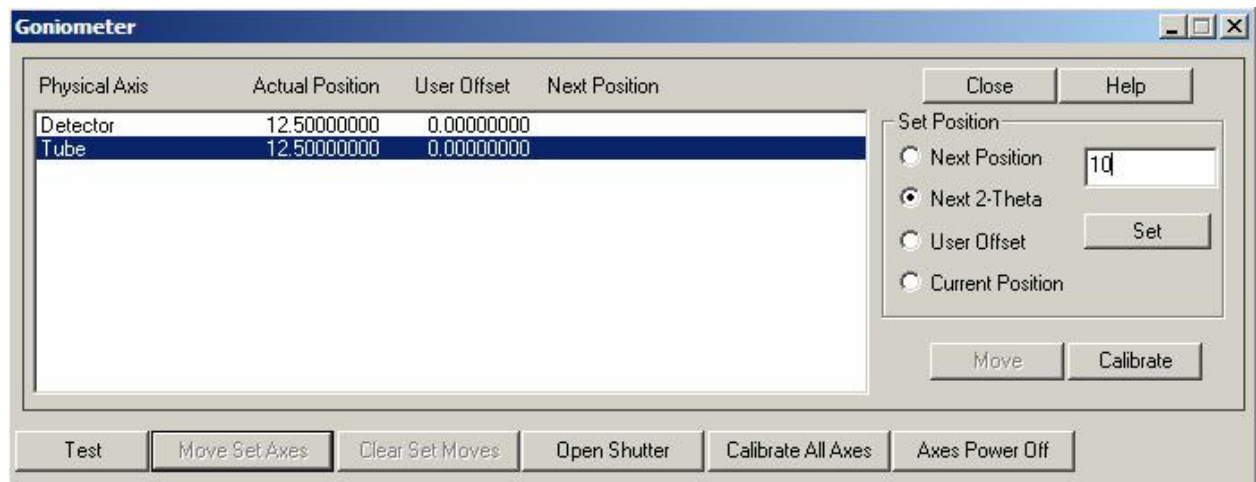
3.12. If you need to work with more samples, you can just create the new event. No need to set goniometer angles to 5.0.

## 4. Shutdown Procedure:

4.1. Hardware → Manual → Goniometer



4.2. Next 2 $\theta$  → Enter '10' → 



4.3. Move Set Axes

4.4. Close

4.5. Log out of computer



4.6. Press green **X-ray Off**

4.7. Press **Control Power On**

4.8. Press red **Water** switch – Light should now be off

4.9. Remove sample and clean up after yourself